

# NATIONAL BUREAU OF STANDARDS REPORT

9615

QUARTERLY REPORT FOR THE PERIOD ENDING JUNE 30, 1967 ON

NBS PROJECT 3120445

INVESTIGATION OF THE DIRECTIONAL EFFECTS  
IN THE STRESS CORROSION OF ALUMINUM ALLOYS

by

Jerome Kruger  
Gilbert M. Ugiansky  
S. Wayne Stiefel  
and  
J.R. Ambrose

for

National Aeronautics and Space Administration  
George C. Marshall Space Flight Center  
Huntsville, Alabama

Contract H-2151A  
Control 1-6-54-01046-01 (1F)



U.S. DEPARTMENT OF COMMERCE  
NATIONAL BUREAU OF STANDARDS

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# NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

NBS REPORT

3120445

September 19, 1967

9615

QUARTERLY REPORT FOR THE PERIOD ENDING 6/30/67

on

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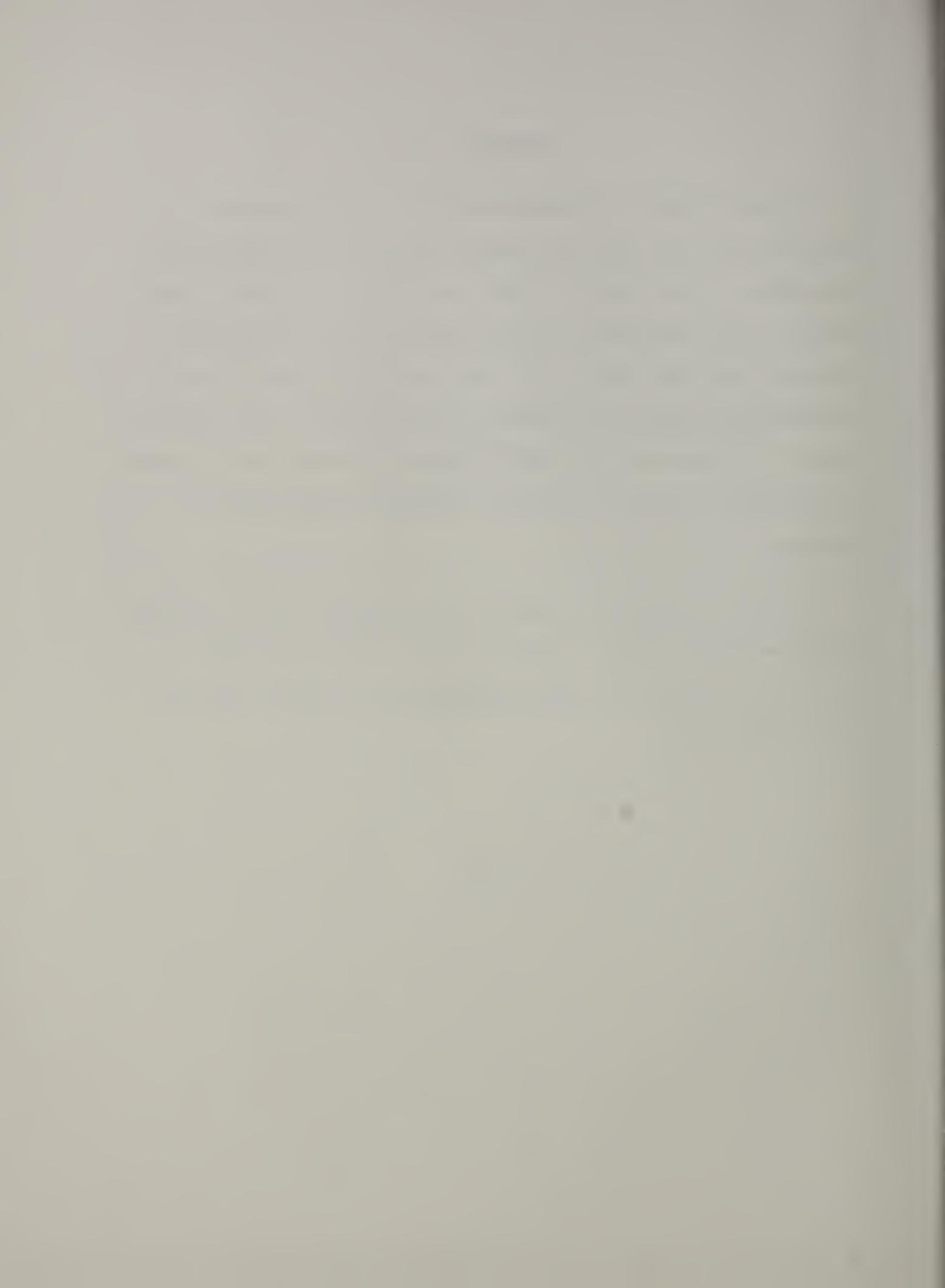
This report was prepared by the Corrosion Section, National Bureau of Standards under Contract No. H-2151A "Investigation of the Directional Effects in the Stress Corrosion of Aluminum Alloys" for the George C. Marshall Space Flight Center of the National Aeronautics and Space Administration. The work was administered under the technical direction of the Propulsion and Vehicle Engineering Laboratory, Materials Division of the George C. Marshall Space Flight Center with D.B. Franklin acting as project manager.



## ABSTRACT

Studies of the role of directionality on stress corrosion susceptibility in the 7075-T651 aluminum alloy have continued during this quarter. Investigation of crack initiation in specimens stressed parallel to the longitudinal axis by scanning electron microscopic techniques have shown conclusively that cracks do initiate virtually instantaneously along grain boundaries oriented normal to the applied stress. It is further shown that the degree of general pitting attack is considerably reduced in specimens undergoing stress corrosion cracking.

Initial electrochemical studies of the corrosion current flowing during the stress corrosion cracking process show that significantly more area is available for crack propagation in the short transverse stressed specimens.

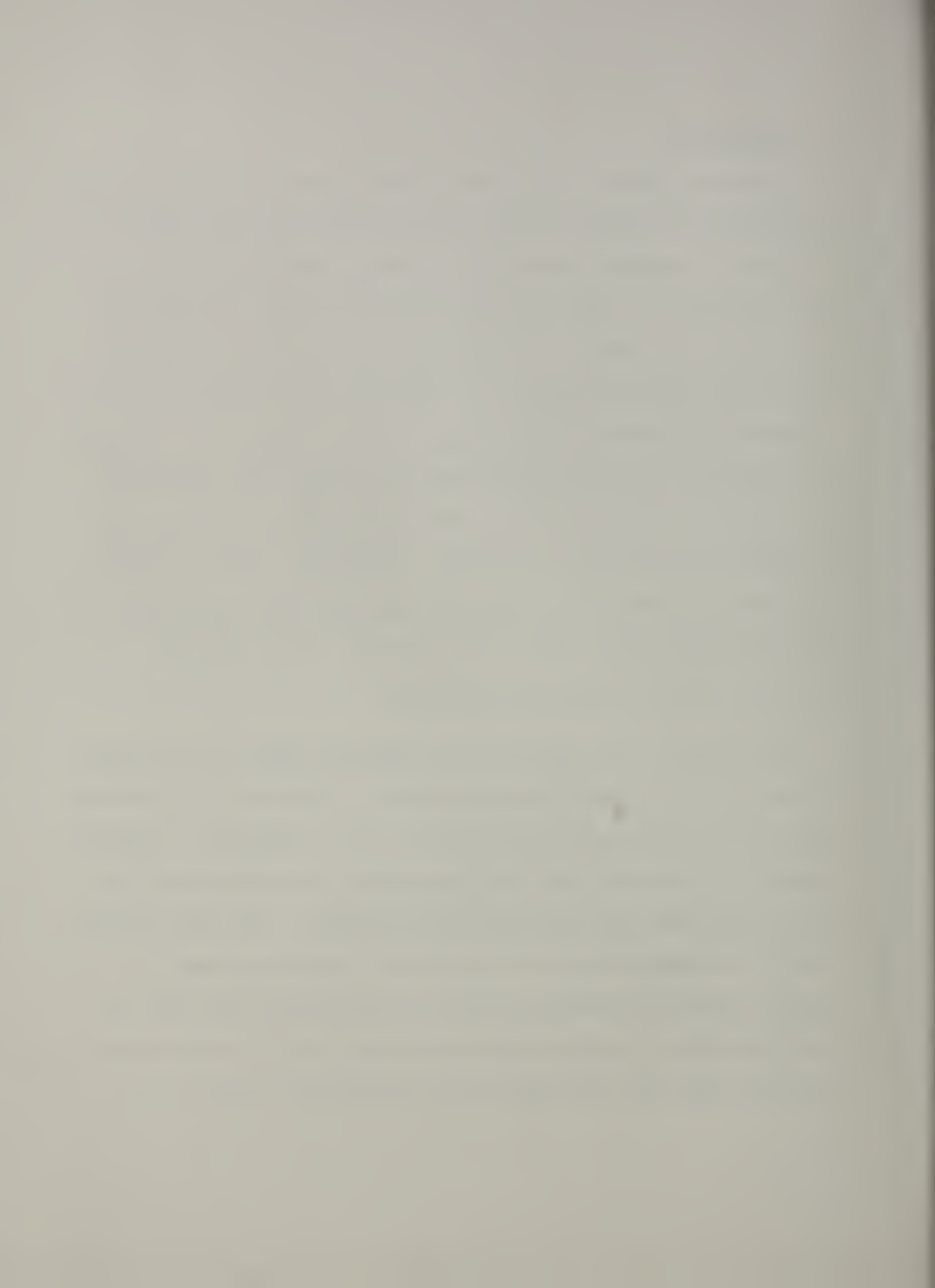




## I. INTRODUCTION

During our studies of the role of directionality on stress corrosion susceptibility in aluminum alloys, we have placed particular emphasis on the effects of preferred orientation, alloying element segregation, and grain morphology. We have shown, that while preferred orientation may be a factor in the stress corrosion susceptibility of material stressed in the short transverse direction, it cannot be used to explain the low susceptibility of material stressed parallel to the grain flow. We have also attempted to correlate stress corrosion cracking with segregation in the 7075-T651 alloy by electron probe microanalysis, but results do not indicate any clear-cut association. On the basis of these observations, further investigation into these areas will not be undertaken during this phase of the project; the last area, the role of grain morphology, however, being actively pursued.

With respect to this area, the last quarterly report indicated that cracking in the 7075-T651 alloy progressed at a faster rate at boundaries normal to the applied stress than parallel to it. Furthermore, cracking appeared to initiate at these normal boundaries in the same short time in both short transverse and longitudinal specimens. This would indicate that the low susceptibility of longitudinally stressed specimens as compared to short transverse specimens is related to the fact that the former has most of its grain boundaries oriented parallel to the stress direction, while the latter has most of its boundaries normal to the

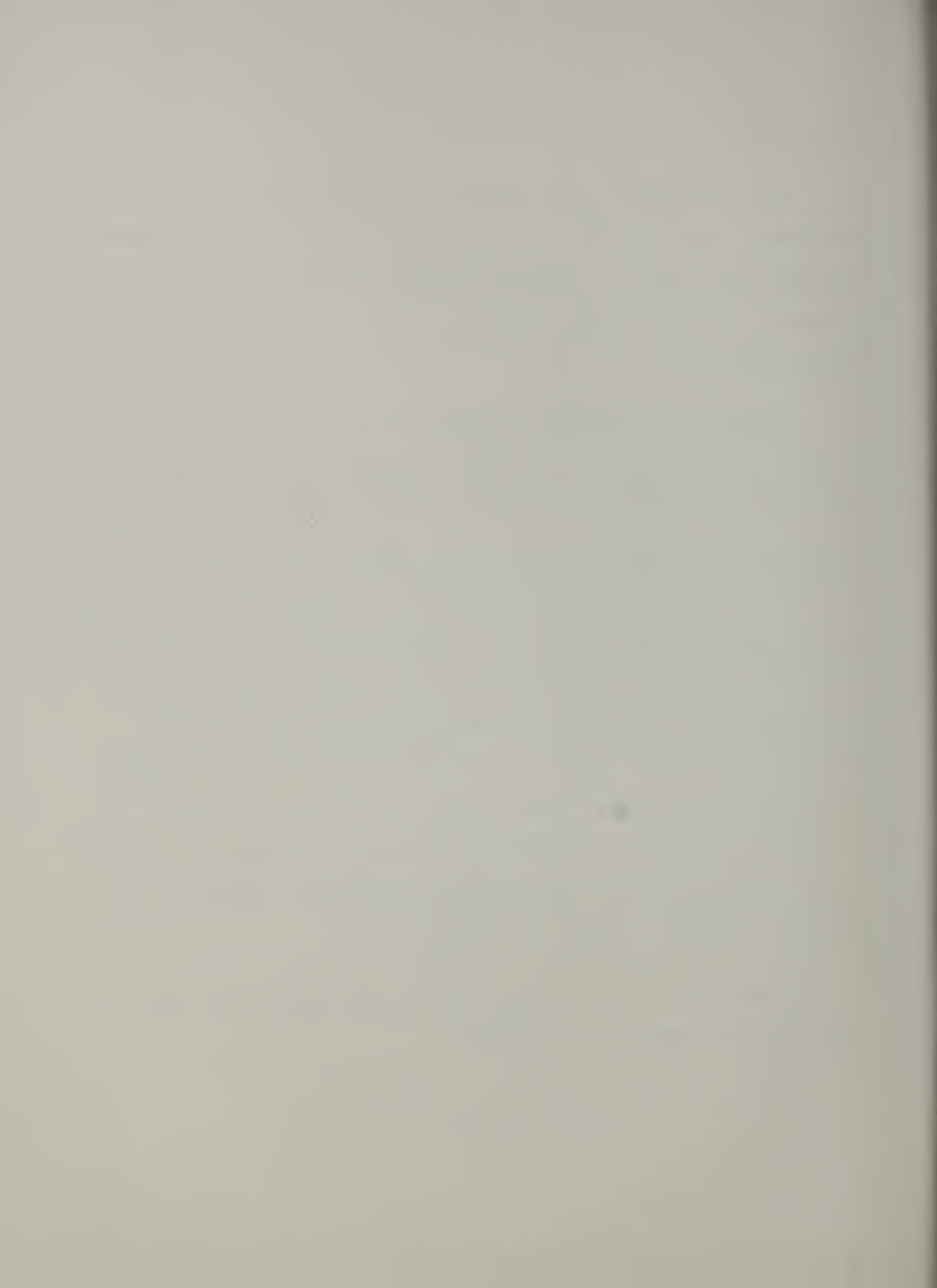


applied stress.

Since there might be some question as to whether the optical micrographs showed intergranular cracking or dissolution, stress corroded surfaces were examined by scanning electron microscopic (SEM) techniques in order to verify the conclusions of the last quarterly report concerning crack initiation.

In addition to the studies of initiation and propagation with respect to directionality, using optical and optical electron techniques, the other major phase of our studies has been electrochemistry. These studies were initiated during this review period. Their purposes are two-fold: 1) to learn something about the precise environment under which cracking occurs, that is, pH, solution composition, and potential, so that some sort of cracking mechanism can be arrived at and related to directionality. In order to investigate the environment for stress cracking, polarization curves were obtained for three systems: chromic acid, sodium chloride and sodium nitrate; 2) to actually measure corrosion rates under stress and without stress with respect to direction and to learn if there is enhancement of corrosion rate depending on direction and stress, as measured by current flow.

During this period, experiments measuring the current for stressed and unstressed specimens were started.



## II. EXPERIMENTAL

### A. Materials & Environments

Material from the 2.5"-thick plate of 7075-T651 aluminum alloy was used for all phases of the investigation presented in this report. Several different stress corrosion environments were also used throughout the investigation. They were:

(1) 0.3% NaCl, 3.0%  $K_2Cr_2O_7$ , 3%  $CrO_3$  (pH = 0.9)

(2) 3.5% NaCl

(3) 1.0 N  $NaNO_3$

with the remainder distilled water in all cases. These solutions will be referred to as the chromate, chloride and nitrate solutions, respectively.

### B. Crack Initiation Studies

The specimen used for the crack initiation studies (Figure 1) was designed specifically to fit into the specimen chamber of a scanning electron microscope. The specimens were extracted from the center of the 7075-T651 plate such that the stressed face was in the short transverse-longitudinal plane and the stress direction parallel to the longitudinal direction. This particular orientation was chosen because it is the grain morphology most immune to stress corrosion cracking, and is ideal for observing crack initiation in an elongated grain structure.

After a final light machine cut, the specimens were polished on



numbers 320, 400 and 600 silicone carbide paper, number 600 microcut, on a magomet\* abraded polishing wheel and finally, electropolished for 30 seconds at 0°C in a solution of 20 ml 70% perchloric acid and 100 ml 95% ethanol at 20 volts and 0.3 amp/cm<sup>2</sup>.

A small strain gage was then attached to the surface of the specimen to be stressed so that the relationship between strain and deflection at the screw could be obtained. After obtaining the strain-deflection curve, the stress-deflection curve was plotted with the help of previously obtained stress-strain curves. Other specimens were then stressed to 75% Y.S. (0.2% offset) (56,500 psi) and masked to have only a 2mm square area exposed (Figure 2) to the corrodent. These specimens were then corroded for various times from 30 seconds to 16 hours in the chromate solution. Surface features were then observed in a scanning type electron microscope.\*\*

### C. Electrochemical Studies

Two specimen designs were used: 1) 0.75 x 0.75 x 0.030-inch square specimens (Figure 3), capable of being stressed parallel to either axis in a given plane; 2) tuning fork specimen (Figure 4a) with a 0.375 x 0.500-inch face. In each instance, specimens were machined from the center portion of the plate in order to eliminate those effects resulting from the variation in stress corrosion susceptibility with plate

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\*Buehler

\*\*Japan Electronic Optics Lab., Ltd. JSM Scanning Type Electron Microscope



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depth.

The procedure for surface preparation of the specimens prior to exposure has been previously mentioned in this report.

Square specimens were clamped in a stainless steel bracket (Figure 3) and all but an 1/8-inch square area in the center of the specimen coated with electroplater's stop-off lacquer. Tuning fork specimens were stressed before mounting in the support bracket and coated with stop-off lacquer. Specimen assemblies were then placed in the appropriate electrochemical cell (Figures 3, 4b, and 5).

Two cell systems were used during the course of the investigation. The initial design consisted of a pyrex glass body with a built-in Luggin capillary and platinized platinum counter electrode. The cell was later modified so that self-stressed tuning fork specimens could be used. In addition, the Luggin capillary of the reference electrode could be positioned to within 0.1mm of the specimen surface to minimize IR drop in the electrolyte. The potential of the specimen surface was maintained at a selected value by means of a potentiostat. Current measurements were then made as a function of specimen potential. During the course of exposure, the corrodent ( $500 \text{ cm}^3$ ) was circulated at a constant flow rate of  $600 \text{ cm}^3$  per minute and directed at the specimen surface to sweep away gas bubbles which would interfere with current measurements.

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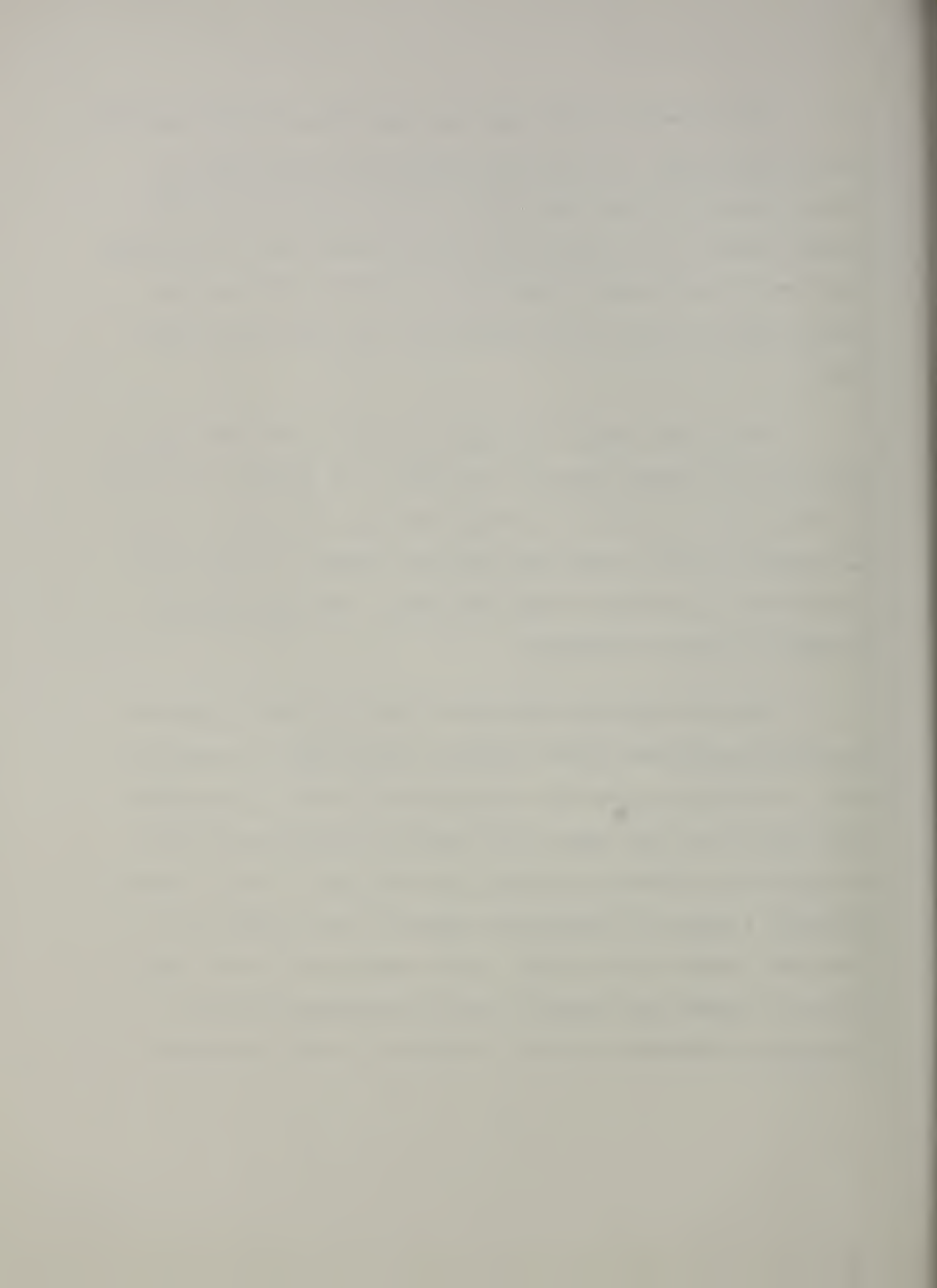
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Electrochemical studies have been made using the three corroducts mentioned previously. The pH of the chromic acid solution was 0.9. Initial experiments showed that excessive film would build up on the specimen surface in the neutral NaCl solution during anodic polarization. Therefore, it was decided to adjust the pH of both the chloride and nitrate solutions to pH 0.9 with hydrochloric and nitric acid, respectively.

Polarization curves were constructed for the 7075-T651 alloy in each of the environments previously described. The potential was varied in both an anodic as well as a cathodic direction, and current readings were made only after a steady state value was reached. At least three sets of data were obtained in each environment. The curves obtained are shown in Figures 6 through 11.

Using the respective polarization curves as guides, potentials have been selected where stress corrosion cracking might be expected to occur. In addition, potentials corresponding to breaks in the polarization curves, where some change in the chemical characteristics of the metal/solution interface is occurring, are being used. Under a constant potential, a tuning fork specimen was exposed to the corroduct for a fixed time interval of two minutes. After exposure, while still under stress, the surface was examined by optical microscopy techniques. During these experiments, we found it necessary to add an alternate



circulation system to the cell where ethanol is used to purge the cell, thereby preventing specimen attack by residual corrodent under open circuit conditions.

Studies have begun on measurement of the corrosion current associated with crack propagation both as a function of time and stress direction. A simple experiment for measuring the current and potential drop across a cell composed of two identical tuning fork specimens immersed in the chromate solution--one stressed to 75% of yield, the other unstressed--has been performed. Current measurements in polarized systems will be made upon completion of the stress corrosion potential studies and arrival of more sensitive detection equipment.

### III. RESULTS & DISCUSSION

#### A. Crack Initiation Studies

Observation of the surface condition of stress corroded specimens utilizing the scanning electron microscope enabled study of the crack initiation process in the 7075-T651 alloy. Cracks were seen to initiate in as short a time as 30 seconds in the longitudinally stressed specimens (Figure 12). Successively longer exposure times produced cracking as shown in Figures 13 through 18. Cracking was found to occur primarily at the ends of the elongated grain boundaries, normal to the applied stress, as shown in Figure 14. This figure also points out the difficulty in studying crack initiation by low magnifications. The same crack can





clearly be seen in Figure 15 at the higher magnification easily obtainable in the scanning electron microscope. Cracking after 7 minutes exposure (Figure 16a and 17a) was confirmed by releasing the stress on the specimens and observing closure of the crack (Figure 16b and 17b). Stress corrosion cracks on the surfaces of specimens exposed for 5 to 7 minutes were seen to have propagated to approximately the same extent as cracks on a specimen exposed for 16 hours (Figure 18).

Cracks were thus seen to initiate in times as short as 30 seconds, indicating almost instantaneous cracking on a specimen stressed parallel to the highly elongated grain structure. In the corrodent used, short transverse specimens will fail in approximately 5 minutes and longitudinal specimens in 24 hours. Since crack initiation is, however, very rapid for the longitudinally stressed specimens, on those boundaries normal to the stress direction, its long time to failure must be due to the fact that crack propagation is difficult because most of the crack path lies along boundaries parallel to the stress direction. Thus, an elongated grain structure would be much more susceptible when stressed normal than when stressed parallel to the principal axis of the grains. It is believed that the threshold stresses for the different grain morphologies (different specimen orientations in a highly elongated grain structure) can be related to the applied stress necessary to crack grain boundaries at various angles to the stress direction. If





this is true, then the threshold stress for short transverse specimens approximates the stress necessary to crack grain boundaries normal to the applied stress and the threshold stress for longitudinal specimens approaches the stress needed to crack boundaries parallel to that stress.

Figures 19 through 21 show surfaces of unstressed specimens exposed for 10 seconds and 5 minutes, respectively. General pitting has occurred to a much greater extent on the unstressed specimens than on the stressed specimens seen earlier (Figures 12 through 18). The stifling of general pitting on specimens undergoing stress corrosion cracking may be due to the stressed boundary areas suitably oriented for cracking to occur becoming considerably more anodic than those anodic areas normally susceptible to pitting attack. The net effect would be inhibition of general corrosion by an internal cathodic protection mechanism.

#### B. Electrochemical Studies

Results from the measurement of open-circuit current flow in the cell composed of a stressed and unstressed specimen were as follows: for a longitudinal stressed specimen, the current density was 20 microamp/cm<sup>2</sup> and was relatively constant over the exposure period. Regular fluctuations were noted, but these could be due to hydrogen evolution which would cause changes in the electrode surface area. Current measured in the short transverse cell system rose steadily from an initial level of 20 microamp/cm<sup>2</sup> to 80 microamp/cm<sup>2</sup> over a 4 minute period until failure



occurred. Since the current is some measure of the amount of surface available for reaction, this experiment shows that the S.T. specimen as expected from the microscopic studies has more of its grain boundaries favorably oriented for such reactions than the longitudinal and shows how it varies with time.

It is well known that specimens of the 7075-T651 alloy exhibit shorter times to failure in the chromate solution, and much less severe general pitting attack, than in the chloride environment. Furthermore, on the basis of the polarization curves for this alloy in these solutions (Figures 6 through 11) it is readily seen that 7075-T651 is considerably more active in the 3.5% NaCl solution. From these observations, and the previously noted stifling of pitting during stress corrosion, it is clear that susceptibility to stress corrosion is not dependent upon general corrosion susceptibility, but in all probability, depends upon stress enhanced breakdown of a passive film (probably at the grain boundary) followed by anodic dissolution at such grain boundaries.

Figures 6 through 11 are the polarization curves for the 7075-T651 alloy in chromate environment, 3.5% sodium chloride, and the 1.0N sodium nitrate solutions. The obtaining of these curves is the first step in our electrochemical studies of the role of directionality on mechanism of cracking. The open circuit potentials are labeled on these curves for the chromate and for the chloride solutions and cracking does occur at these potentials.

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Future work will look at the role of directionality and potential at which the surface is held with regard to cracking susceptibility. Optical and electron microscopic studies will be made to see what sort of morphological changes occur at these potentials above the open circuit where cracking is known to occur.

The anodic nitrate curve shows a definite break, and this can be interpreted as being related to the formation of a passive film. This break occurs at approximately +400 mV vs SCE and, as this curve shows, is a more well-behaved system. Efforts will be concentrated on looking at the role of film breakdown and directionality for this system where chloride ions can be added in small increments. This way, the onset of breakdown can then be related to cracking.

## CONCLUSIONS

1. Observations of cracking in as short a time as 30 seconds at grain boundaries normal to the stress on the surface of longitudinally stressed specimens show conclusively that it is the slow rate of crack propagation along boundaries parallel to the applied stress that accounts for the low susceptibility of longitudinal type specimens.

2. Pitting corrosion occurred to a much greater extent on unstressed than on stressed specimens undergoing stress corrosion cracking. We explained this phenomenon by what we termed an "internal cathodic protection mechanism."

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3. Open circuit current flow was significantly higher in a cell composed of an unstressed and stressed short transverse specimen than in a longitudinally stressed cell system. This result agrees with the visual observation of the grain morphology--that is, the resulting surface area is greatest in the short transverse stressed specimen.

#### FUTURE WORK

##### Role of Stress Direction

Since we have suggested earlier that the threshold stresses for the different specimen orientations in an elongated grain structure can be directly related to the threshold stresses for the cracking of grain boundaries at various angles to the stress direction, we plan to determine the threshold stresses for cracking of grain boundaries as a function of their angle with the applied stress.

##### Electrochemical Studies

We plan to continue our studies into the relationship between applied stress level and the electrochemistry of the 7075-T651 alloy with respect to grain morphology. This will be done by:

1) continuing investigations of the relation between passive film breakdown in chloride-nitrate solutions and stress corrosion susceptibility in longitudinal and short transverse directions.

2) optical and/or electron microscopic studies of the metal surface after exposure at various applied stress levels for fixed time intervals under constant potential.





3) corrosion current measurements under potentiostatic conditions corresponding to given chemical processes (i.e.--film breakdown) as a function of applied stress level and direction.

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# PROGRAM PLANNING CHART

## Program

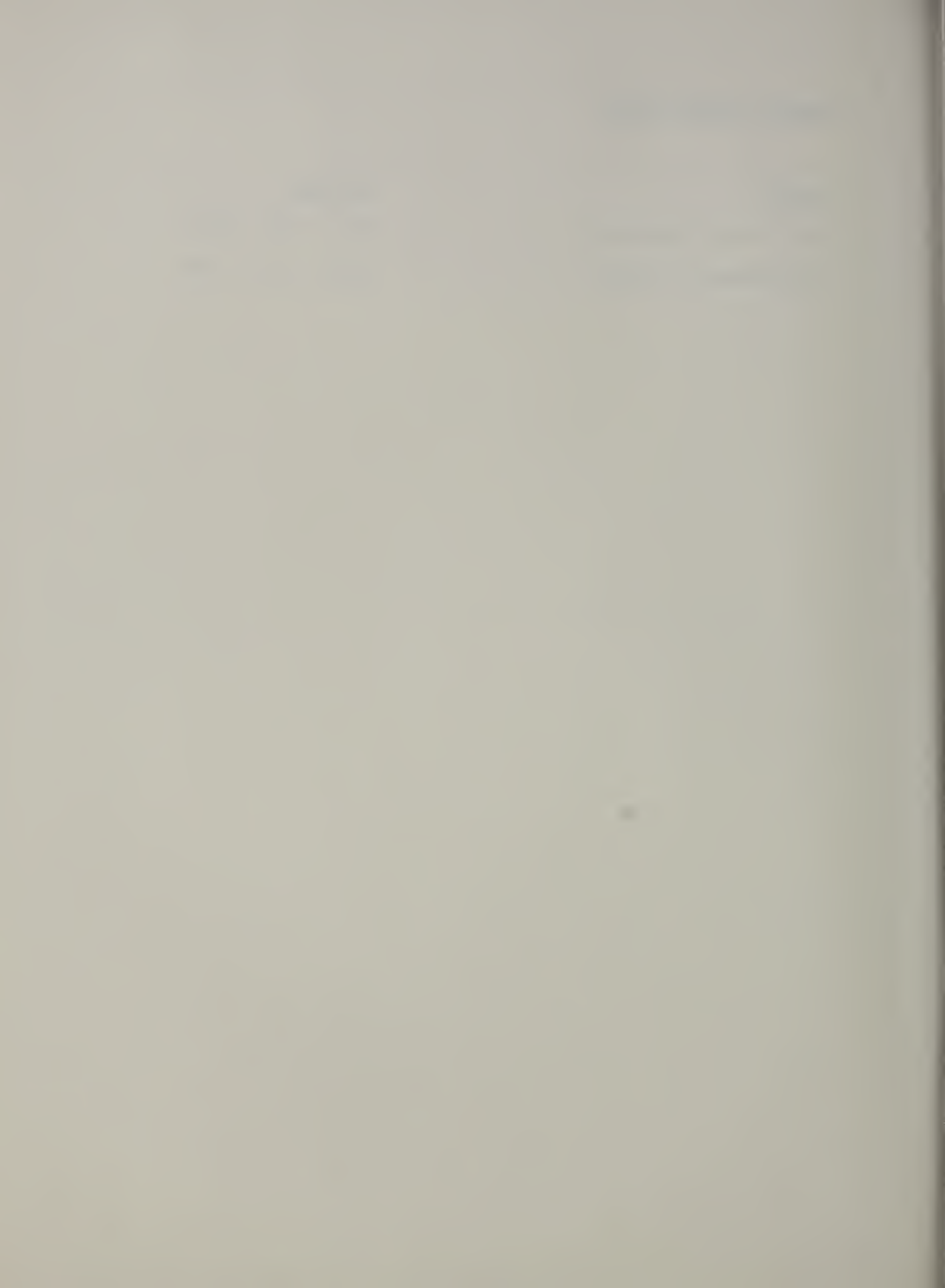
Role of Grain Morphology

Electrochemical Studies

## Duration

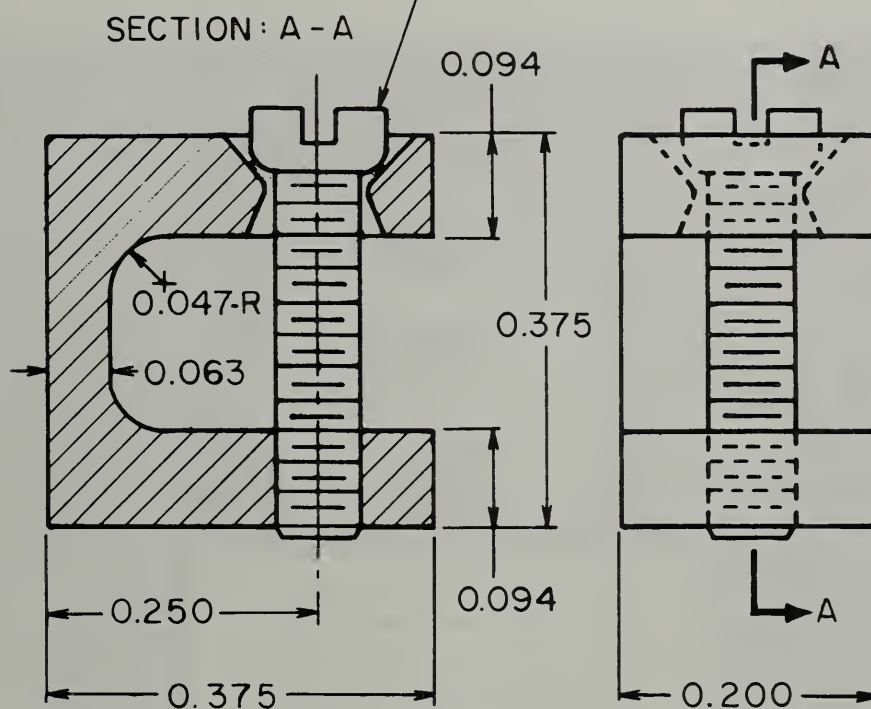
Jan. - Dec., 1967

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# MINIATURE TUNING FORK STRESS-CORROSION SPECIMEN

3-48 MACHINE SCREW  
(WITH SHOULDER ROUNDED)



ALL DIMENSIONS IN INCHES  
TOLERANCE  $\pm 0.001$  THROUGHOUT

Figure 1. Miniature specimen design used in scanning electron microscope studies.



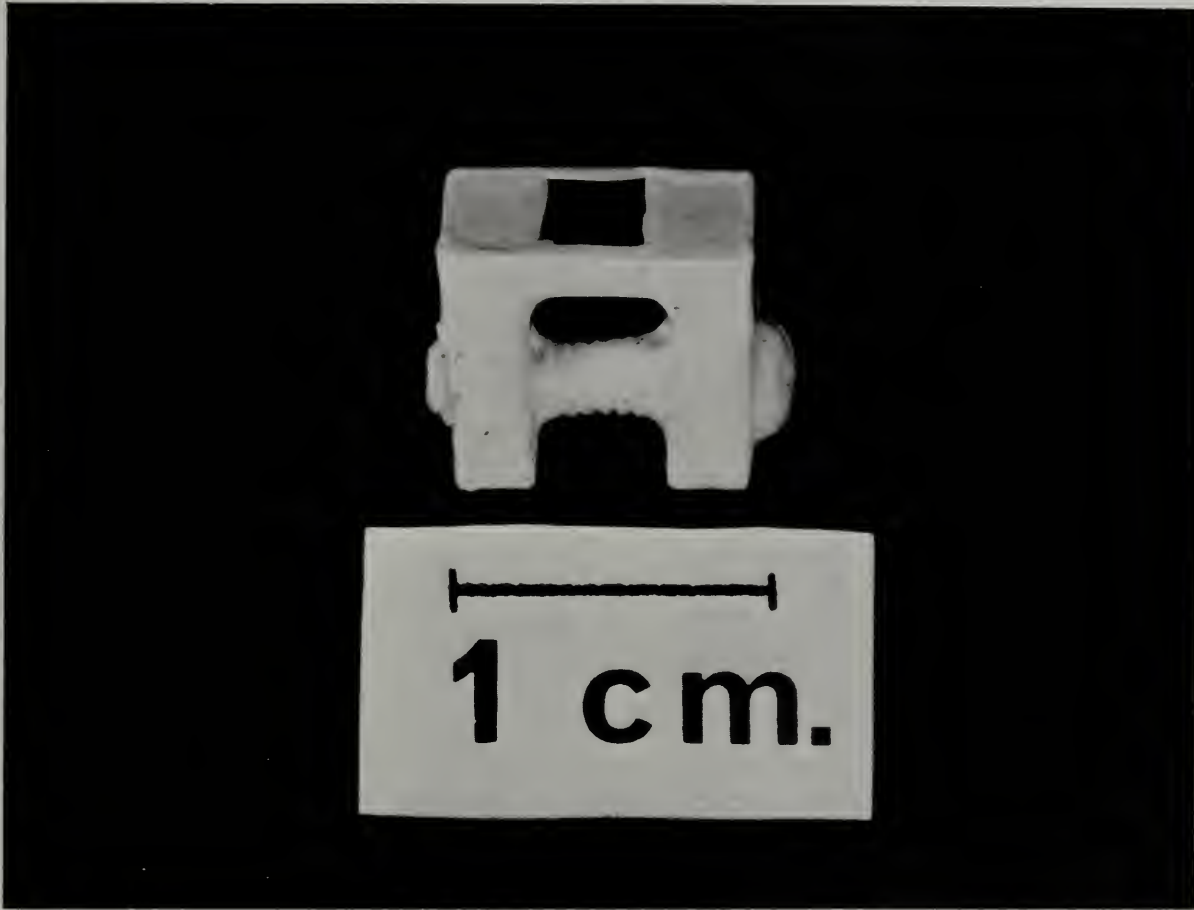


Figure 2. Masked-off miniature tuning fork specimen.



100-5-1



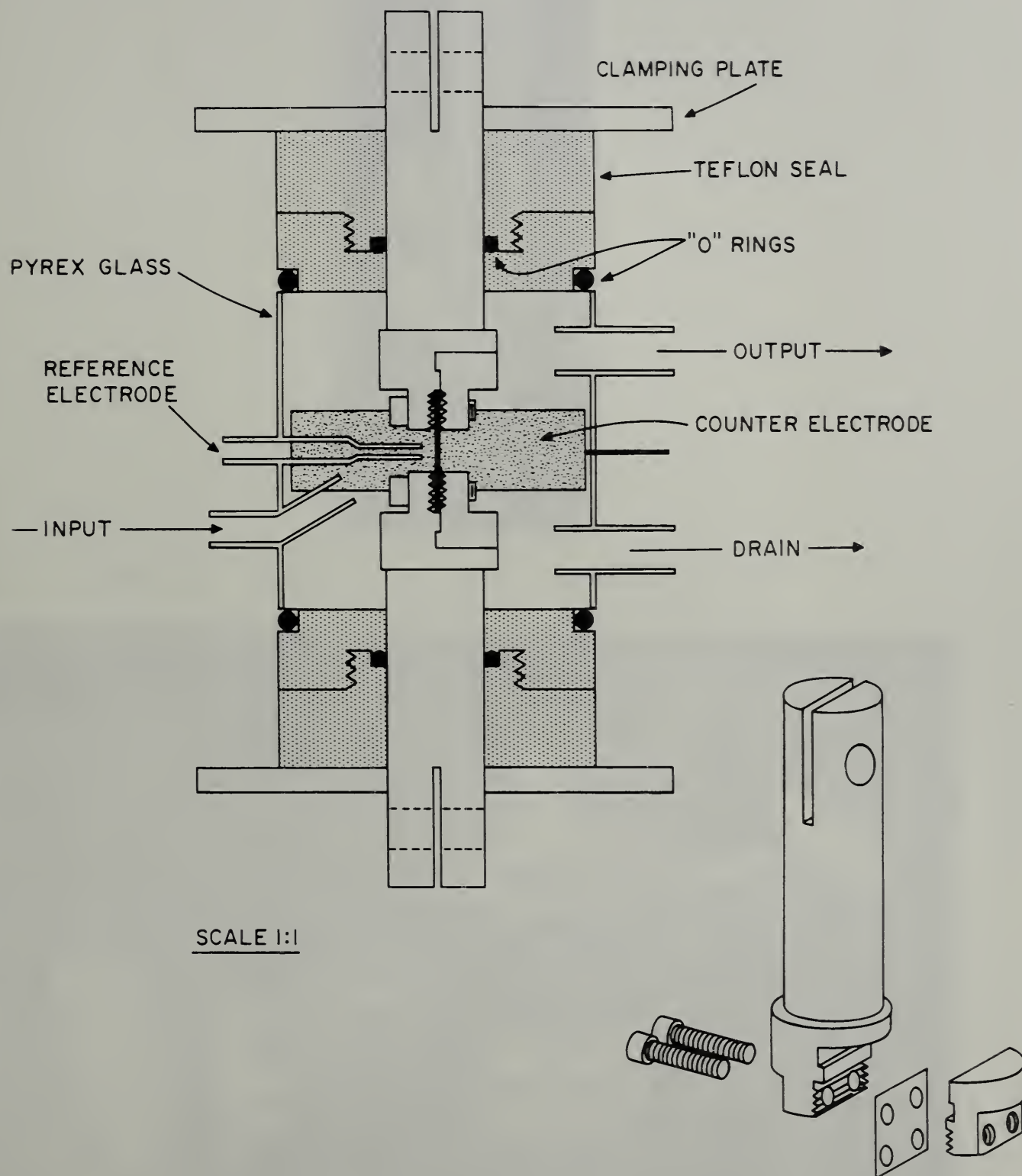


Figure 3. Initial electrochemical stress corrosion cell design for externally applied stress on square specimens.



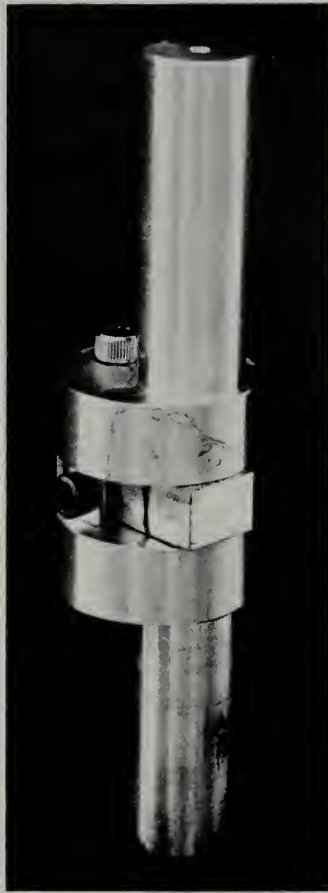


Figure 4a. Tuning fork specimen in mounting bracket.

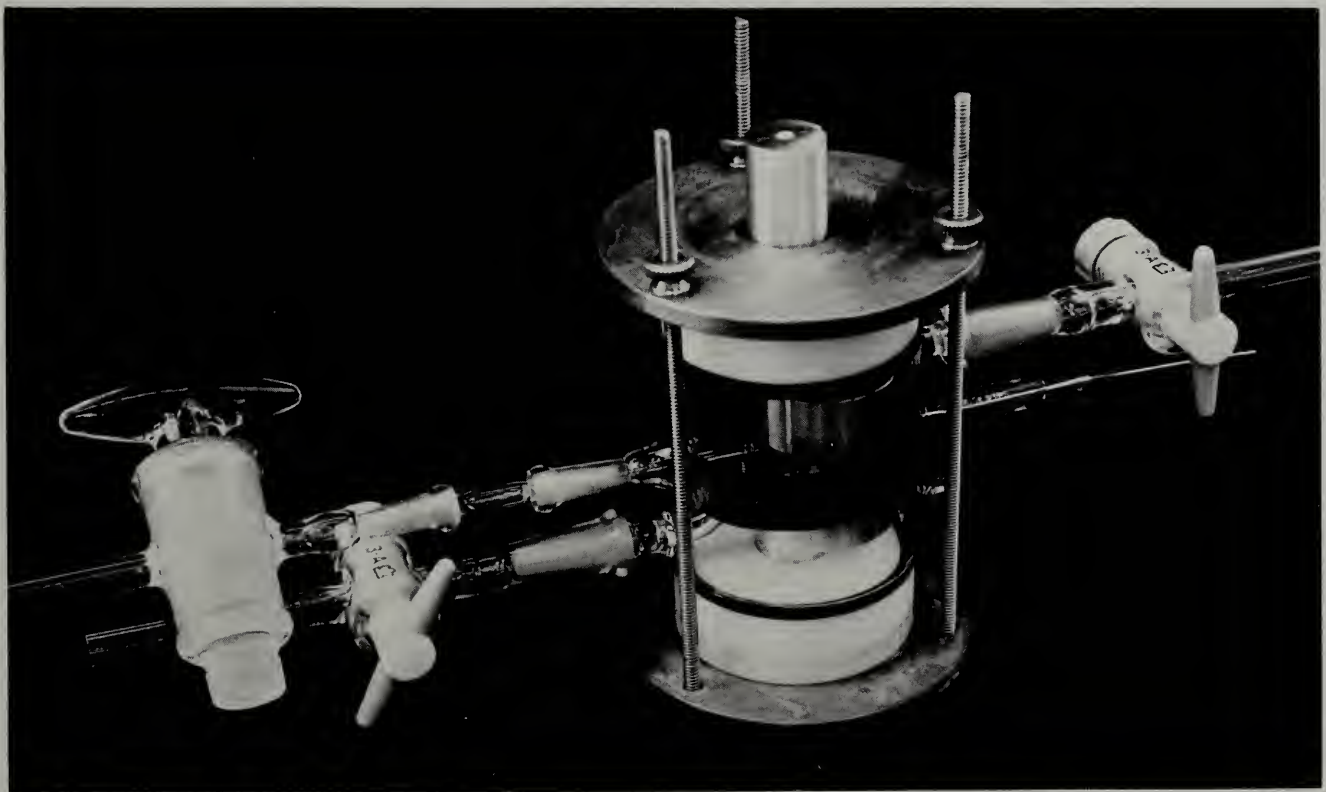


Figure 4b. Assembled electrochemical stress corrosion cell for self-stressed tuning fork specimens.



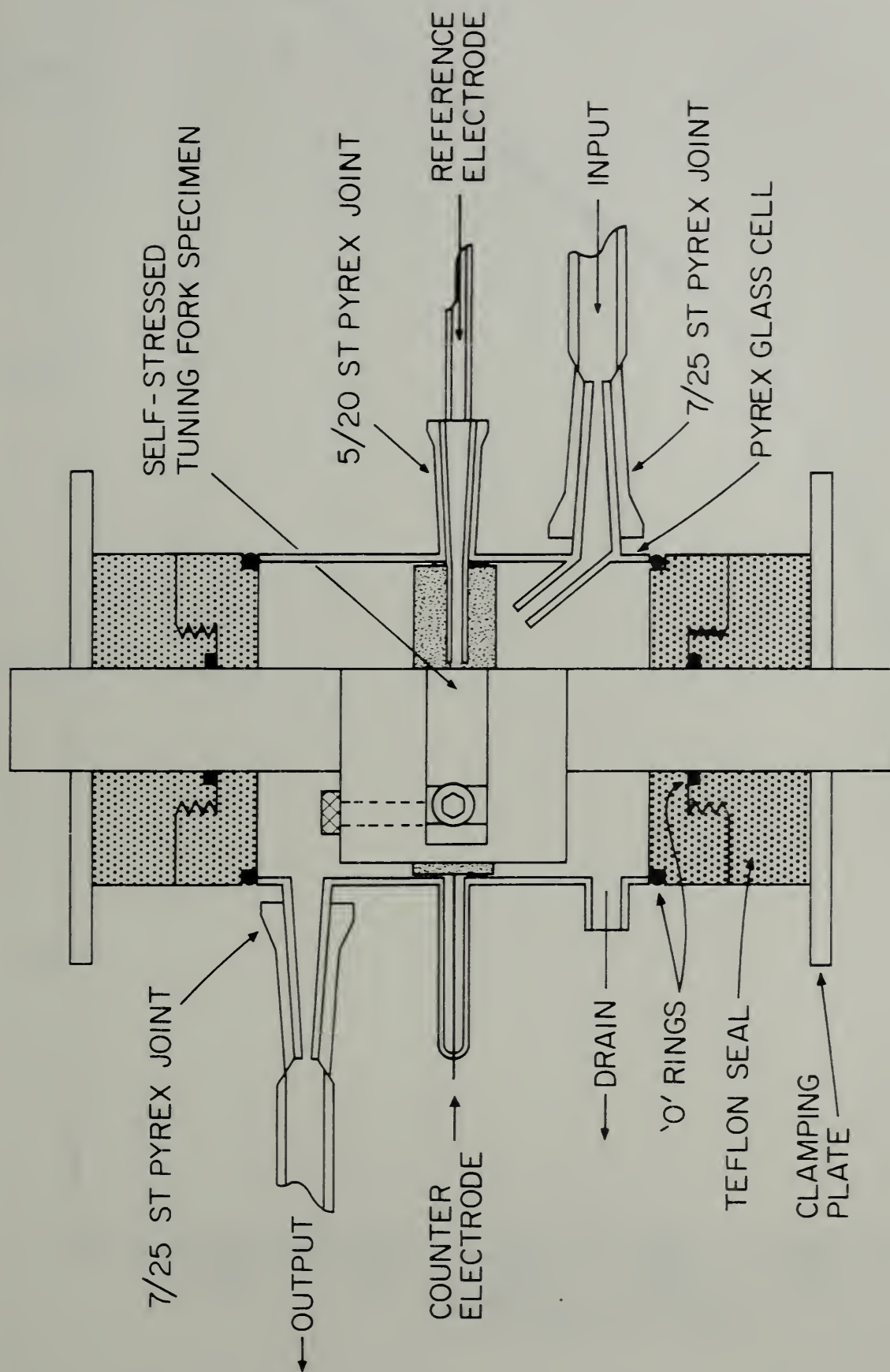


Figure 5. Final electrochemical stress corrosion cell design for self-stressed tuning fork specimens.



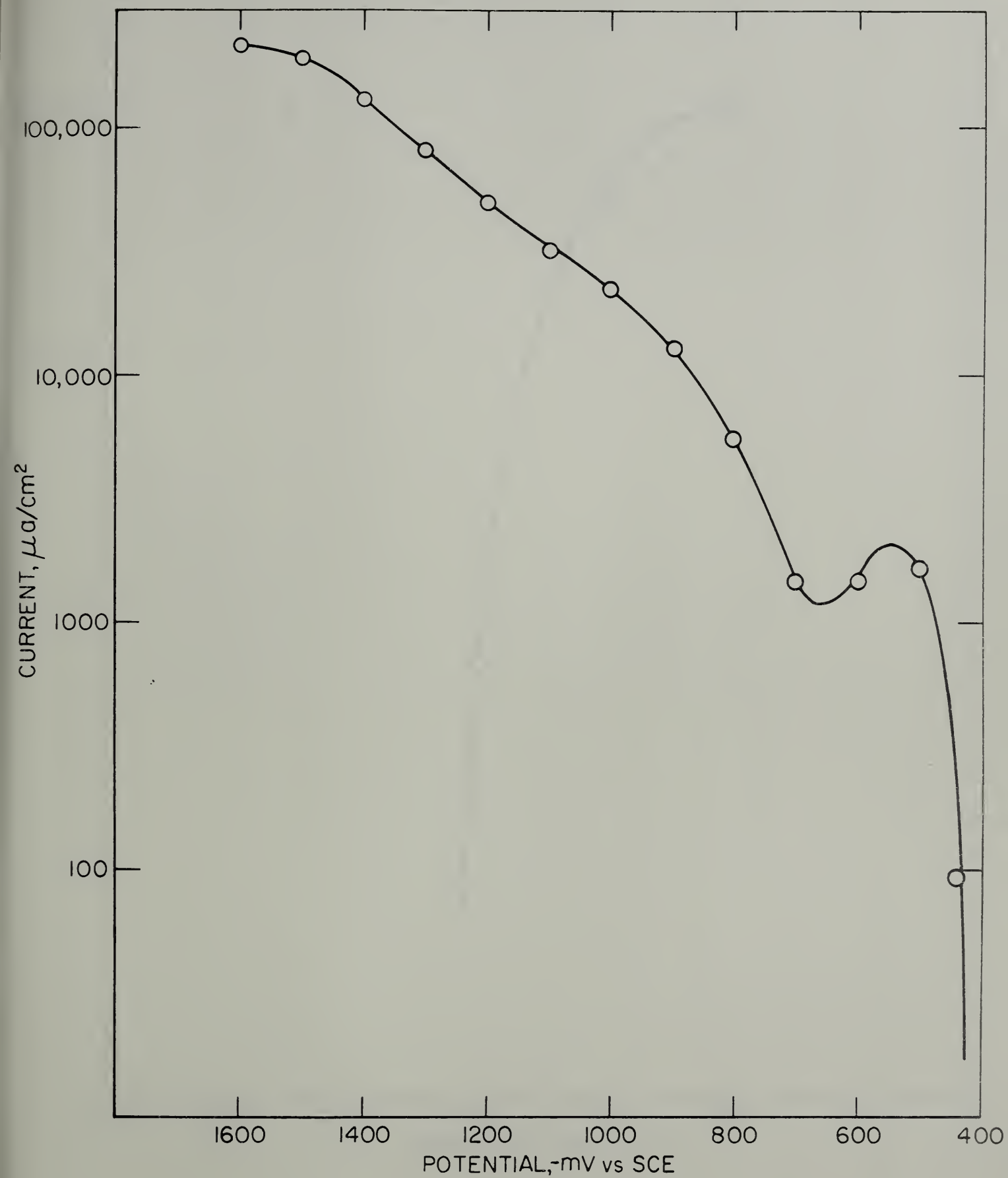


FIGURE 6. CATHODIC POLARIZATION CURVE FOR 7075-T651 IN 0.3% NaCl - 3.0%  $K_2Cr_2O_7$  - 3.0%  $CrO_3$ . OPEN CIRCUIT EMf = -440 mV vs SCE.





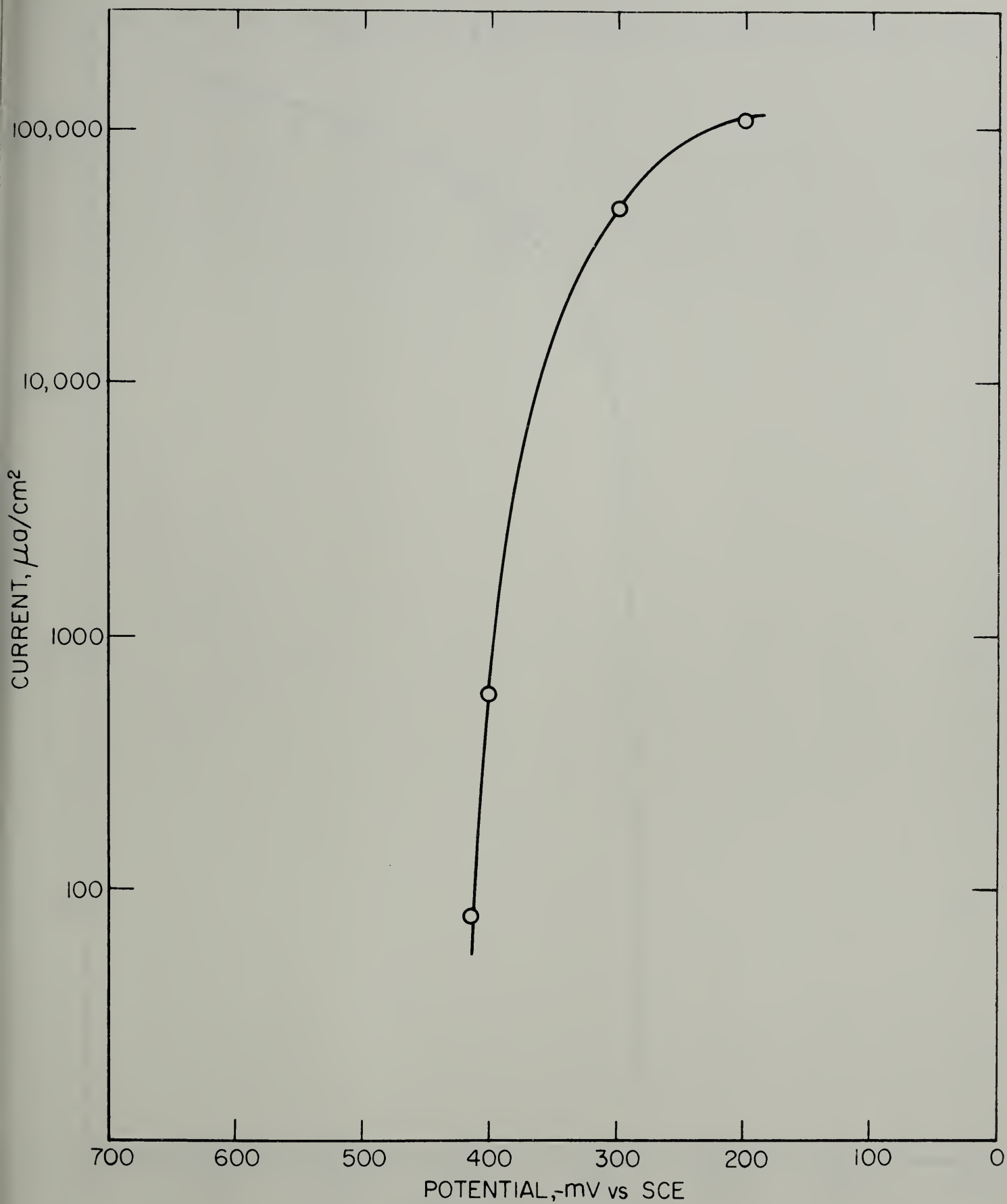


FIGURE 7. ANODIC POLARIZATION CURVE FOR 7075-T651 IN 0.3% NaCl - 3.0%  $\text{K}_2\text{Cr}_2\text{O}_7$  - 3.0%  $\text{CrO}_3$ .



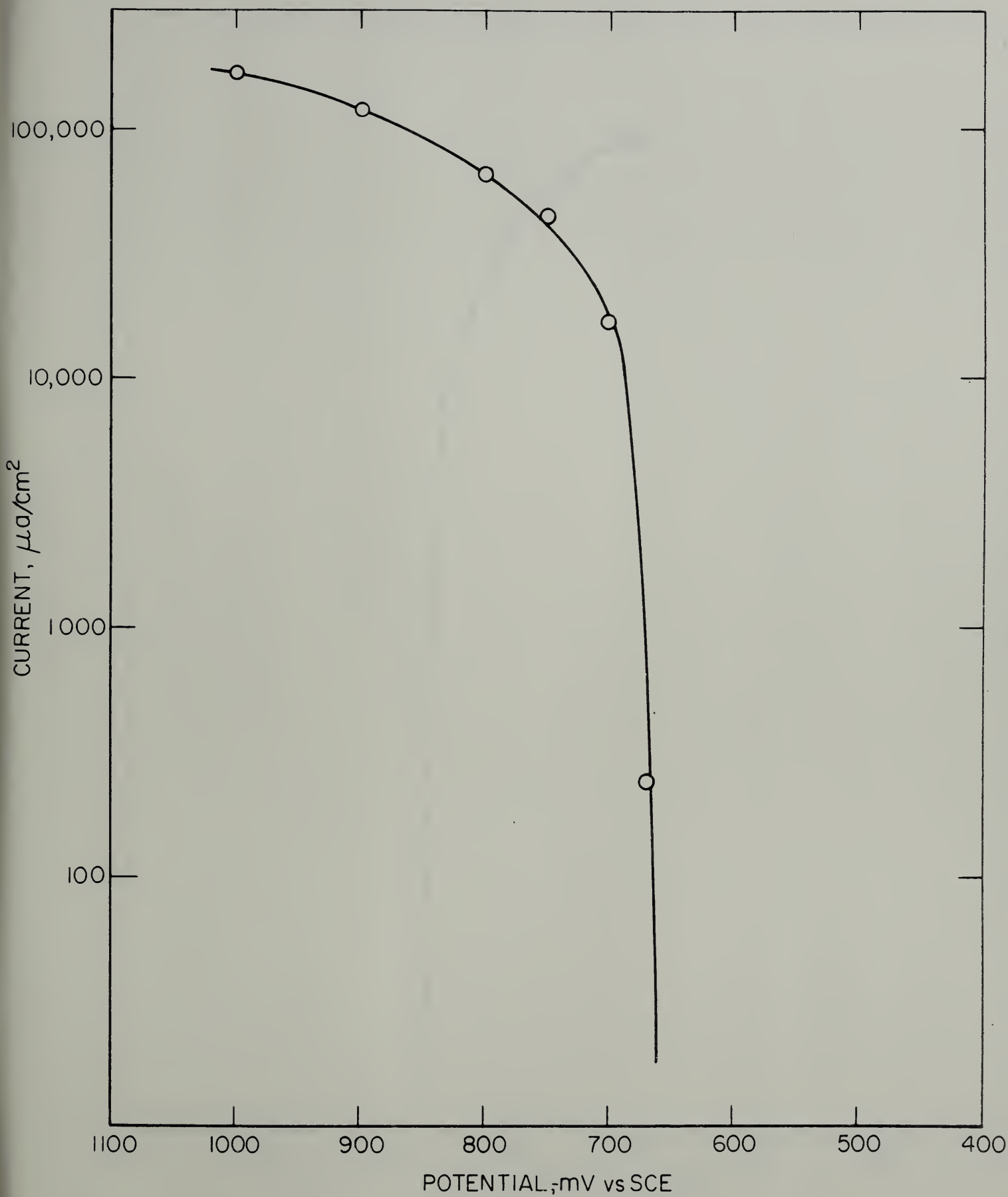


FIGURE 8. CATHODIC POLARIZATION CURVE FOR 7075-T651 IN 3.5% NaCl (pH = 0.9 WITH HCl). OPEN CIRCUIT EMf = -670 mV vs SCE.



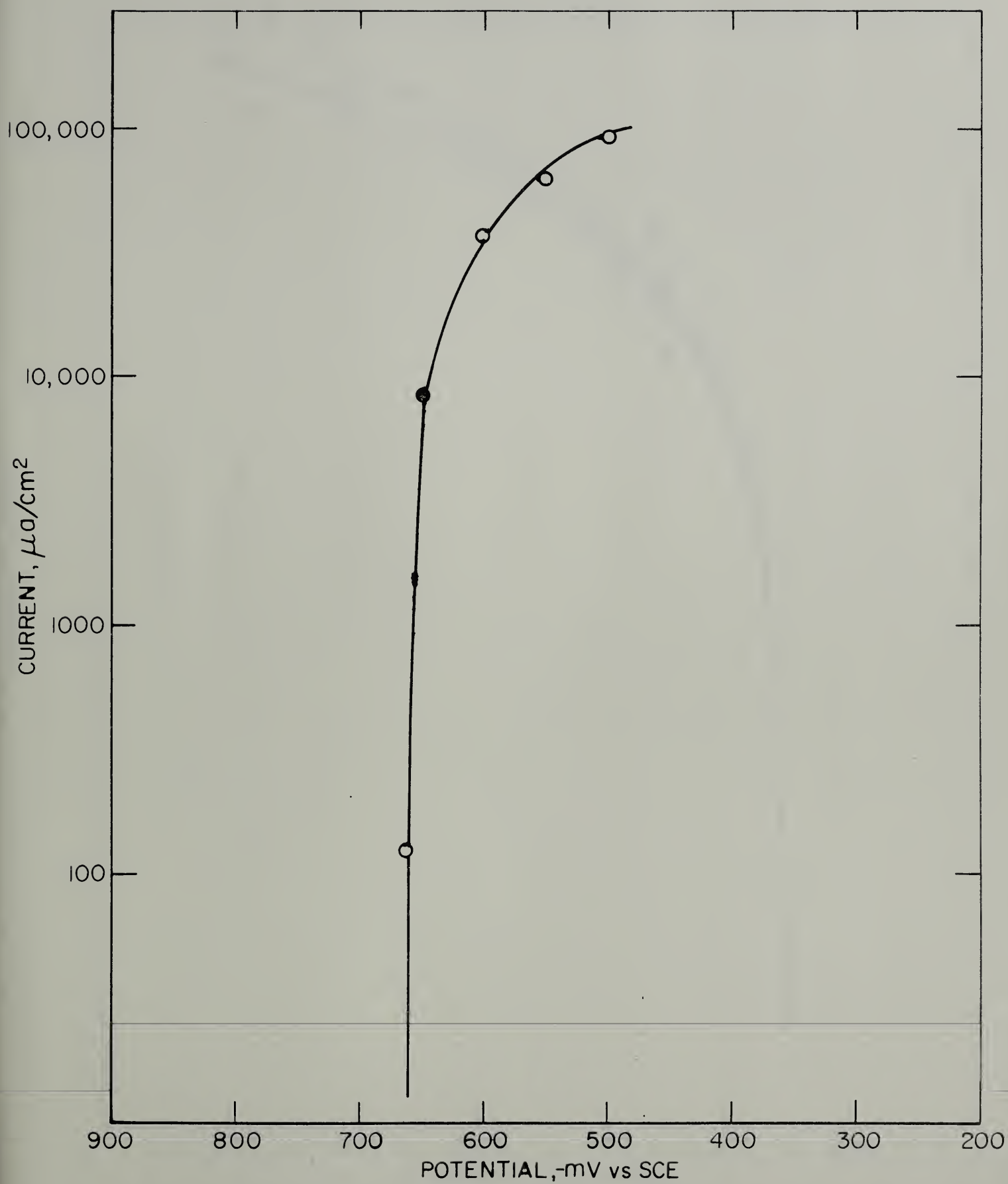


FIGURE 9. ANODIC POLARIZATION CURVE FOR 7075-T651 IN 3.5% NaCl (pH = 0.9 WITH HCl).





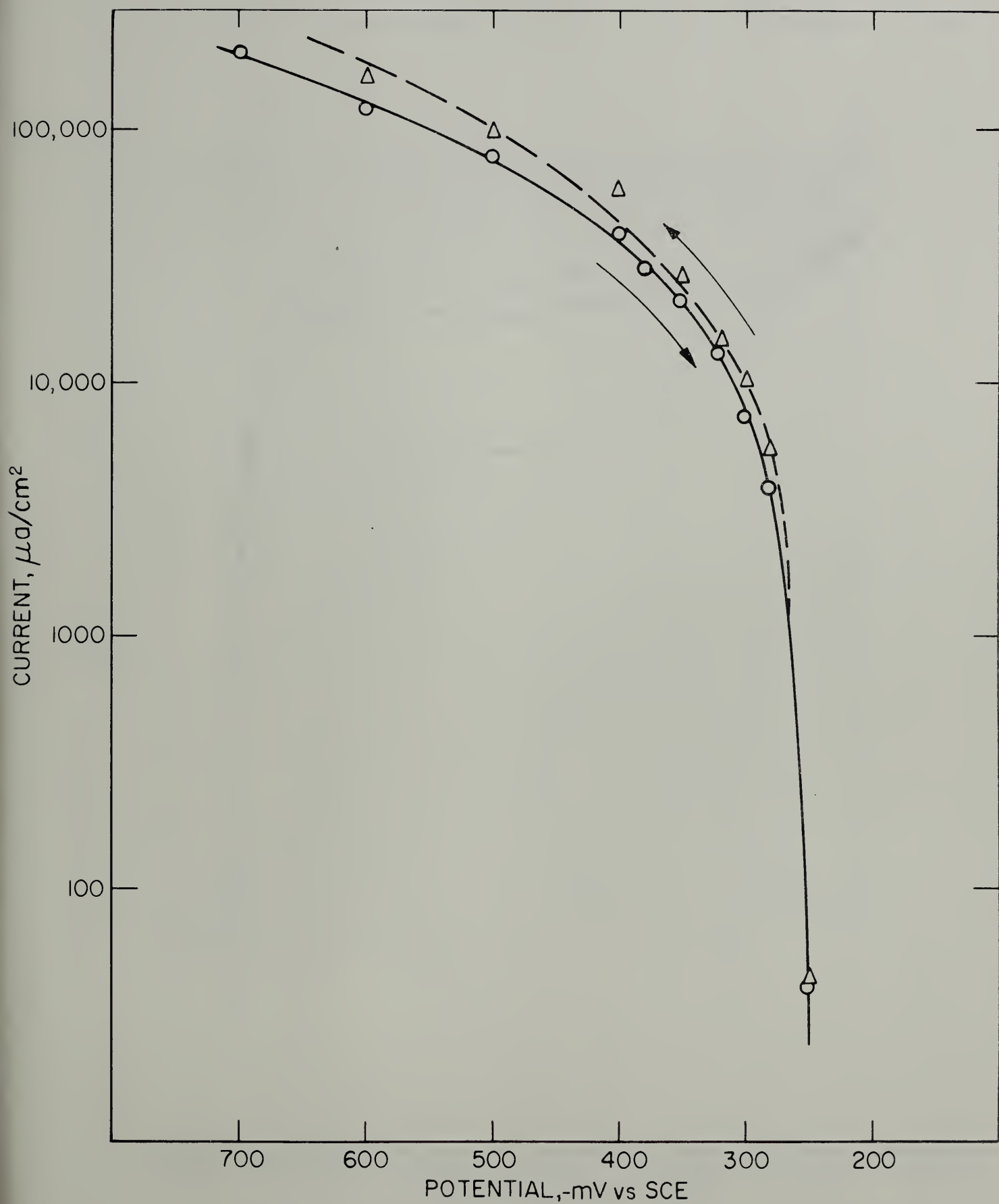


FIGURE 10. CATHODIC POLARIZATION CURVE FOR 7075-T651 IN 1.0N  $\text{NaNO}_3$  (pH = 0.9 WITH  $\text{HNO}_3$ ). OPEN CIRCUIT EMF = -260 mV vs SCE.



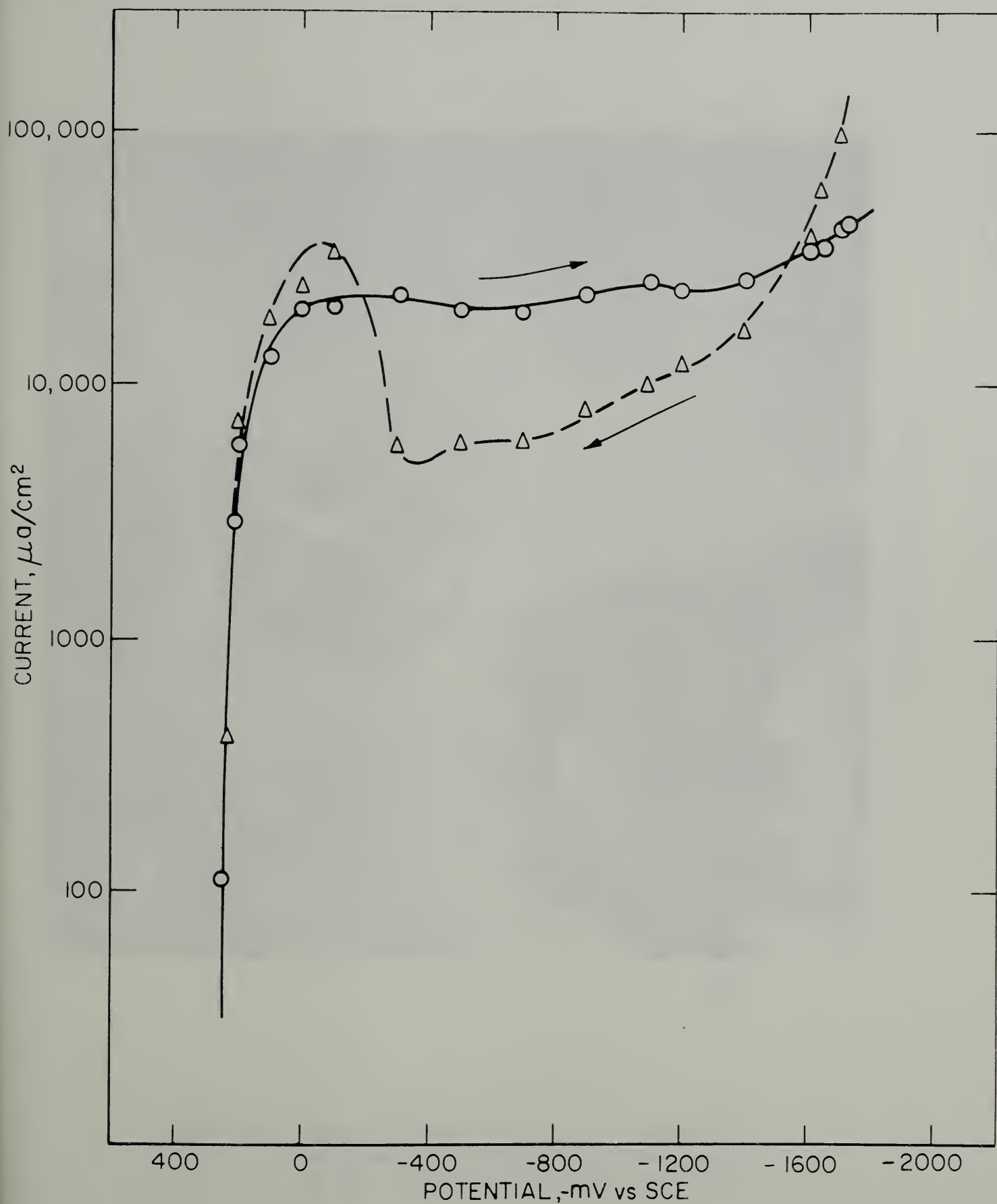


FIGURE 11. ANODIC POLARIZATION CURVE FOR 7075-T651 IN 1.0N  $\text{NaNO}_3$  (pH = 0.9 WITH  $\text{HNO}_3$ ).



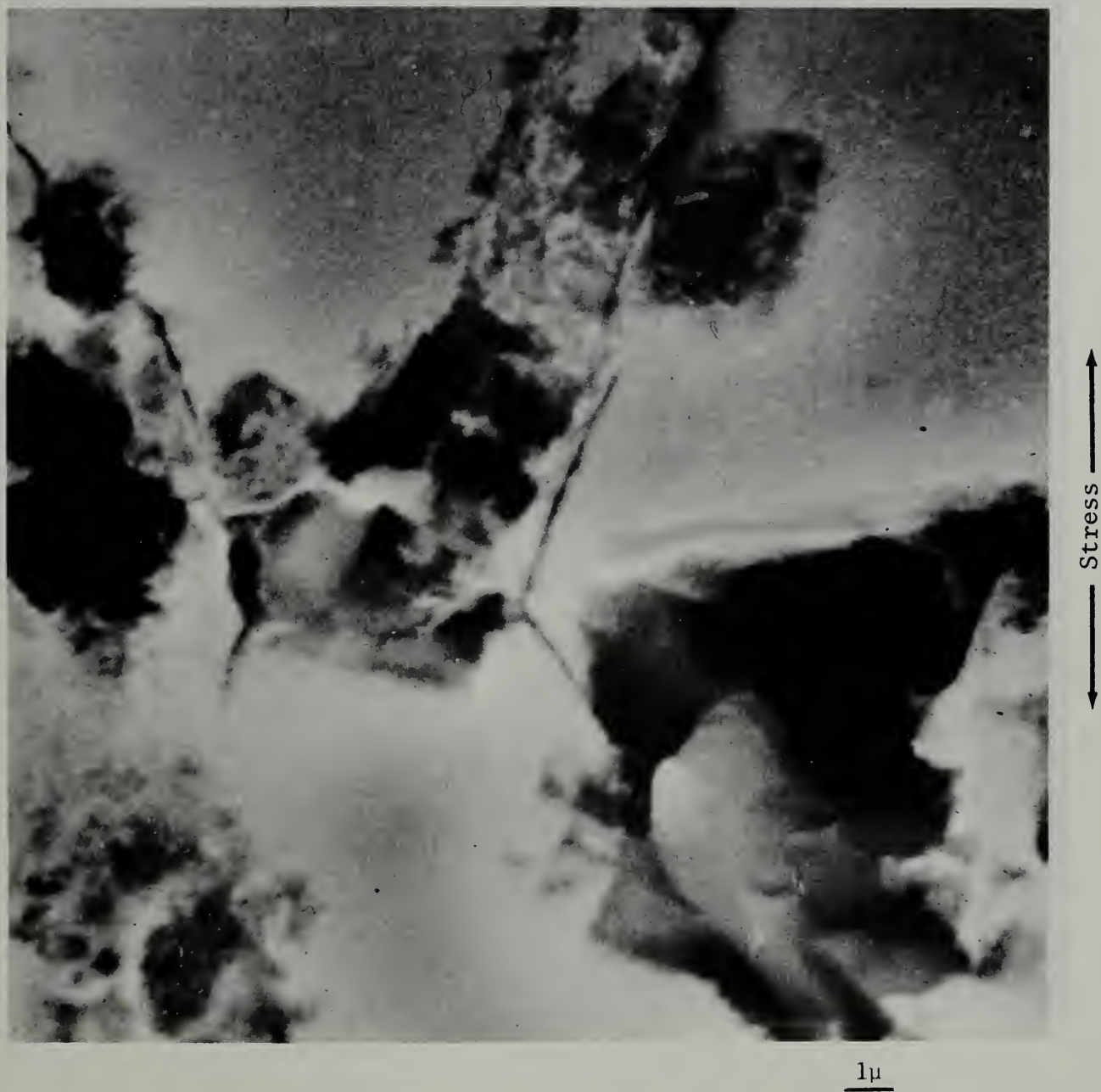
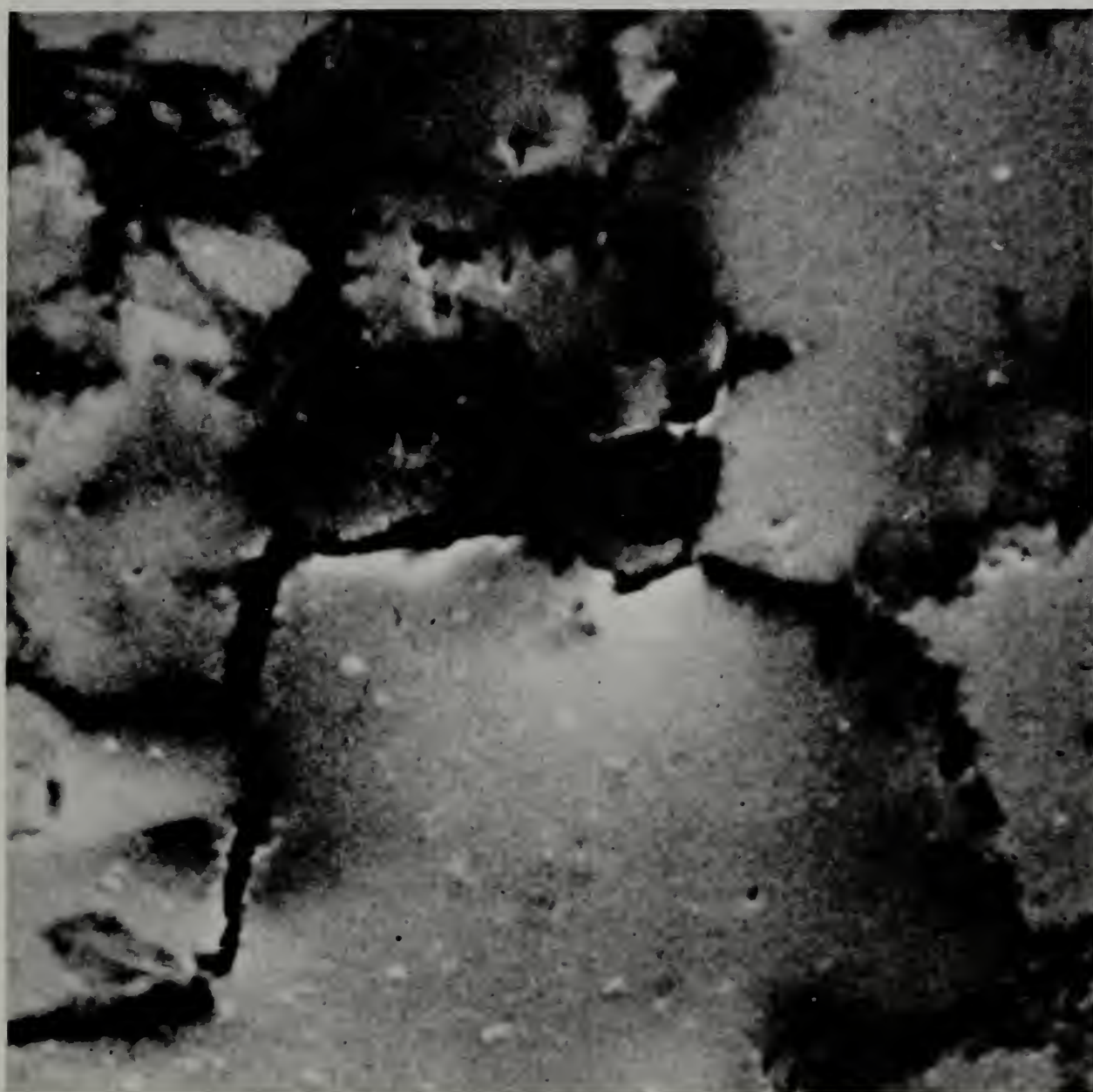


Figure 12. SEM micrograph showing S.C. cracks in longitudinally stressed specimens after 30 sec. exposure in chromate solution. X7,500







↑ Stress ↓

1μ

Figure 13. SEM micrograph showing S.C. cracks in longitudinally stressed specimen after 2 min. exposure in chromate solution. X15,000





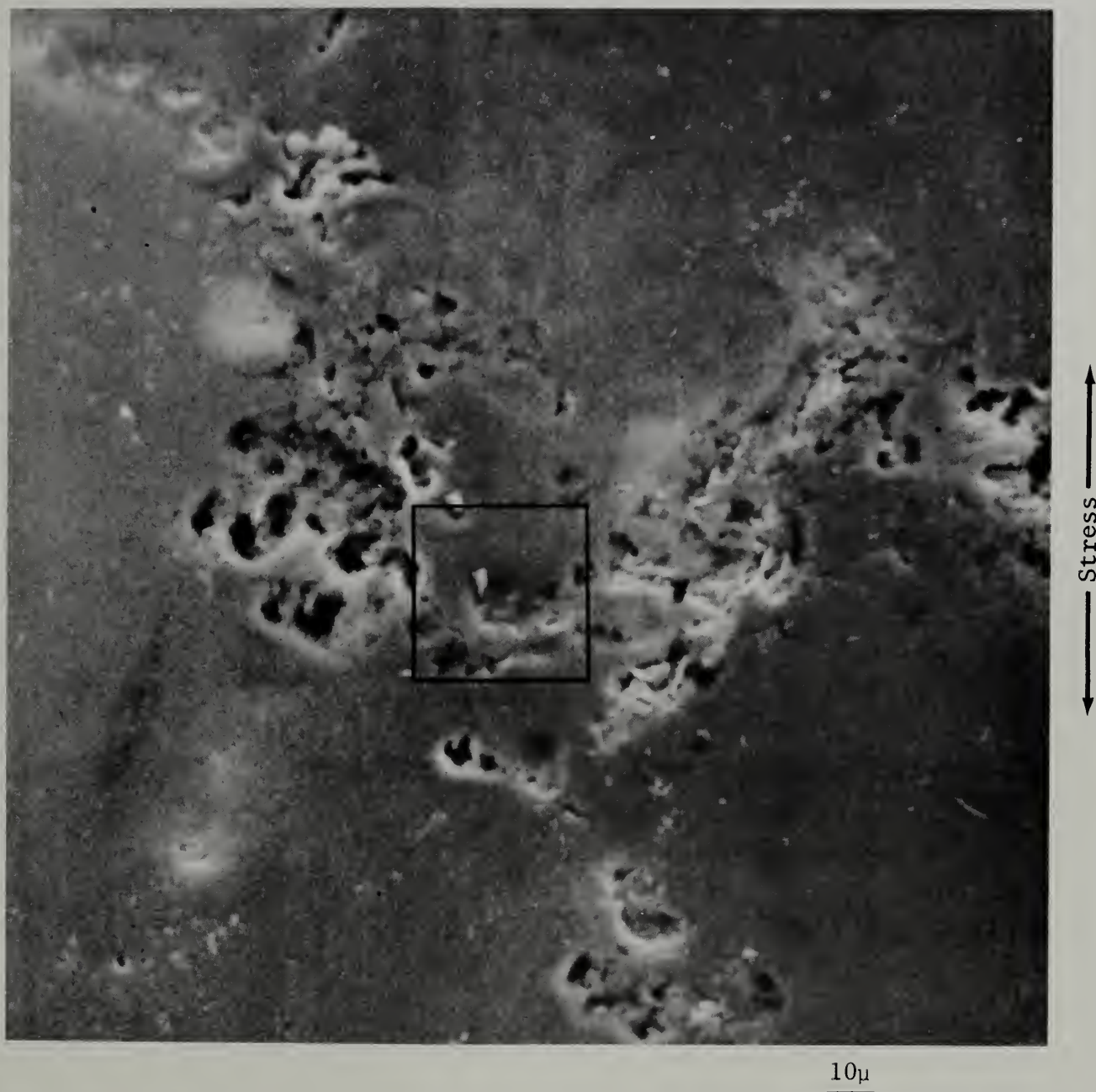


Figure 14. SEM micrograph showing difficulty of locating S.C. cracks at lower magnifications on longitudinally stressed specimen after 5 min. exposure. Note cracking at the end of the elongated grain where the boundary is normal to the applied stress. X750



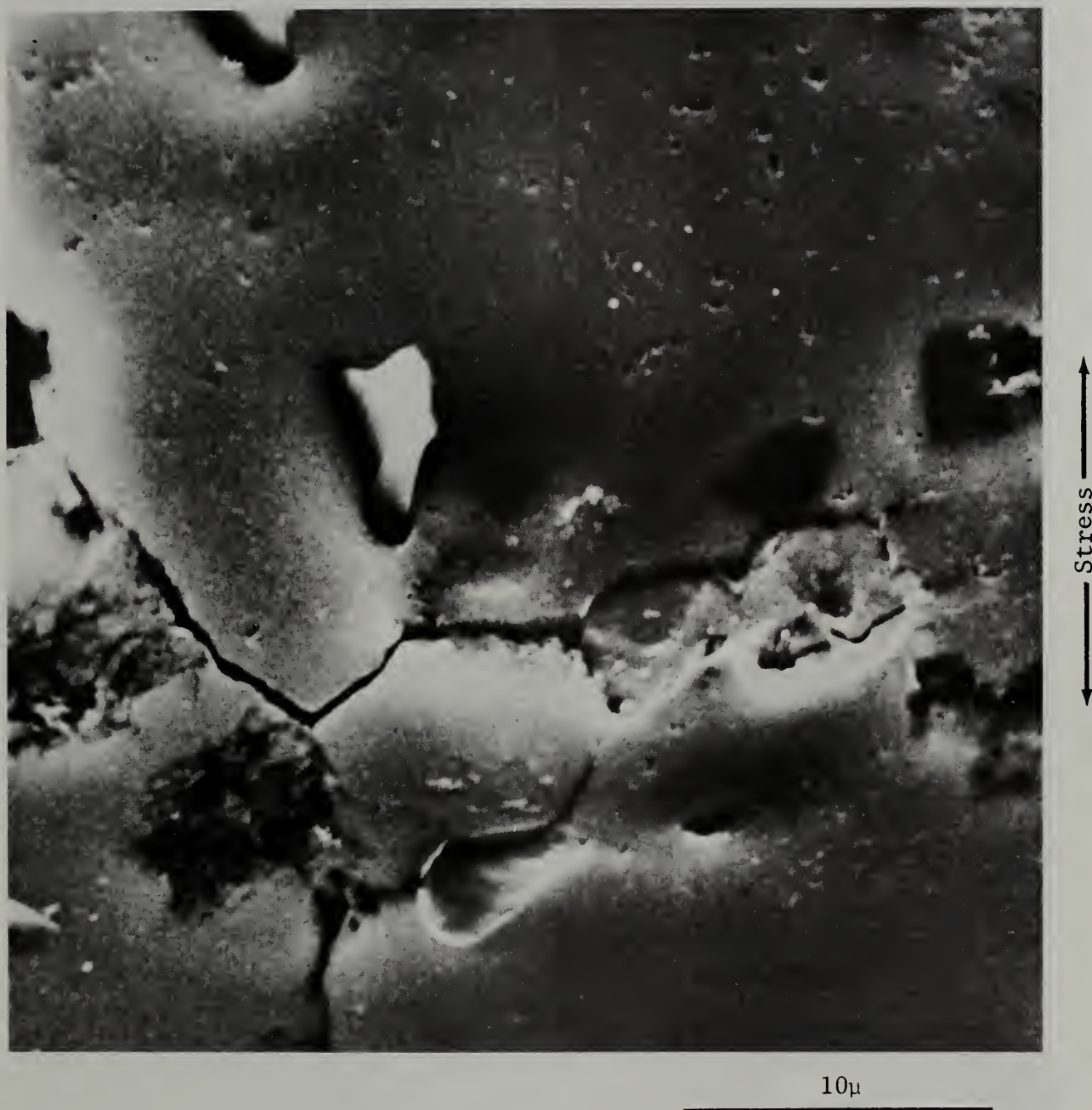


Figure 15. SEM micrograph of area marked in Figure 14. Note cracking at the end of the elongated grain where the boundary is normal to the applied stress. X4,500

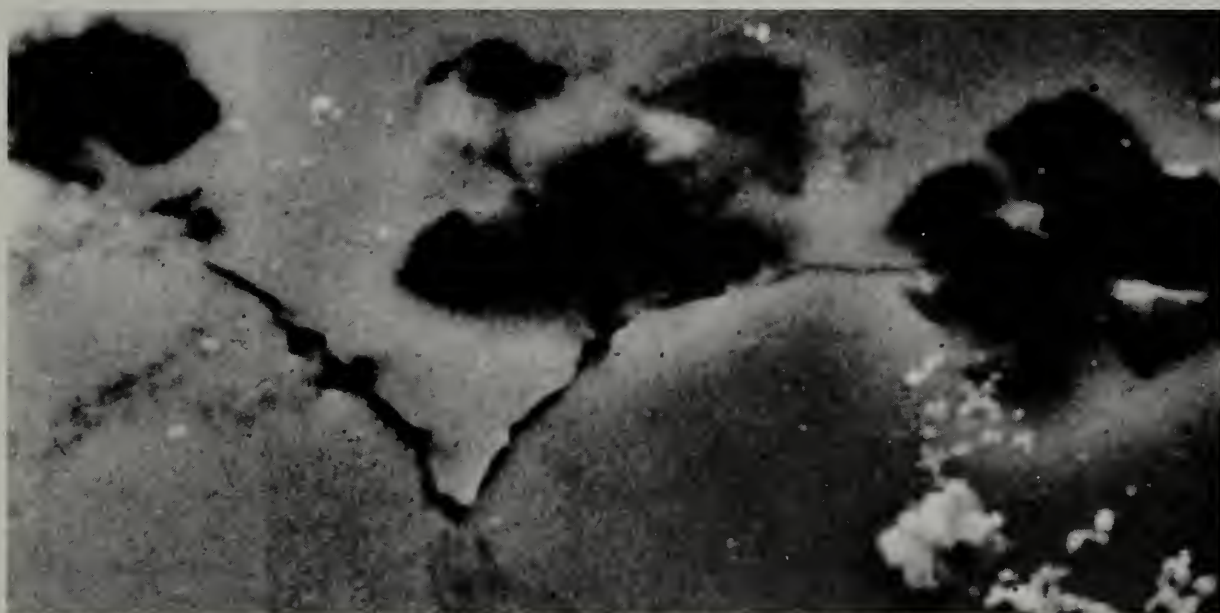






(a)

↑ Stress ↓



(b)

10 $\mu$

Figure 16. (a) SEM micrograph of a longitudinally stressed specimen after 7 min. exposure in chromate solution. X4,500  
 (b) Micrograph of same area after releasing the stress on the specimen. Note closure of the crack. X4,500





(a)

↑ Stress ↓



(b)

1μ

Figure 17. (a) SEM micrograph of same area as Figure 16 after 7 min. exposure in chromate solution. X15,000  
(b) Micrograph of same area after releasing the stress on the specimen. Note closure of the crack. X15,000





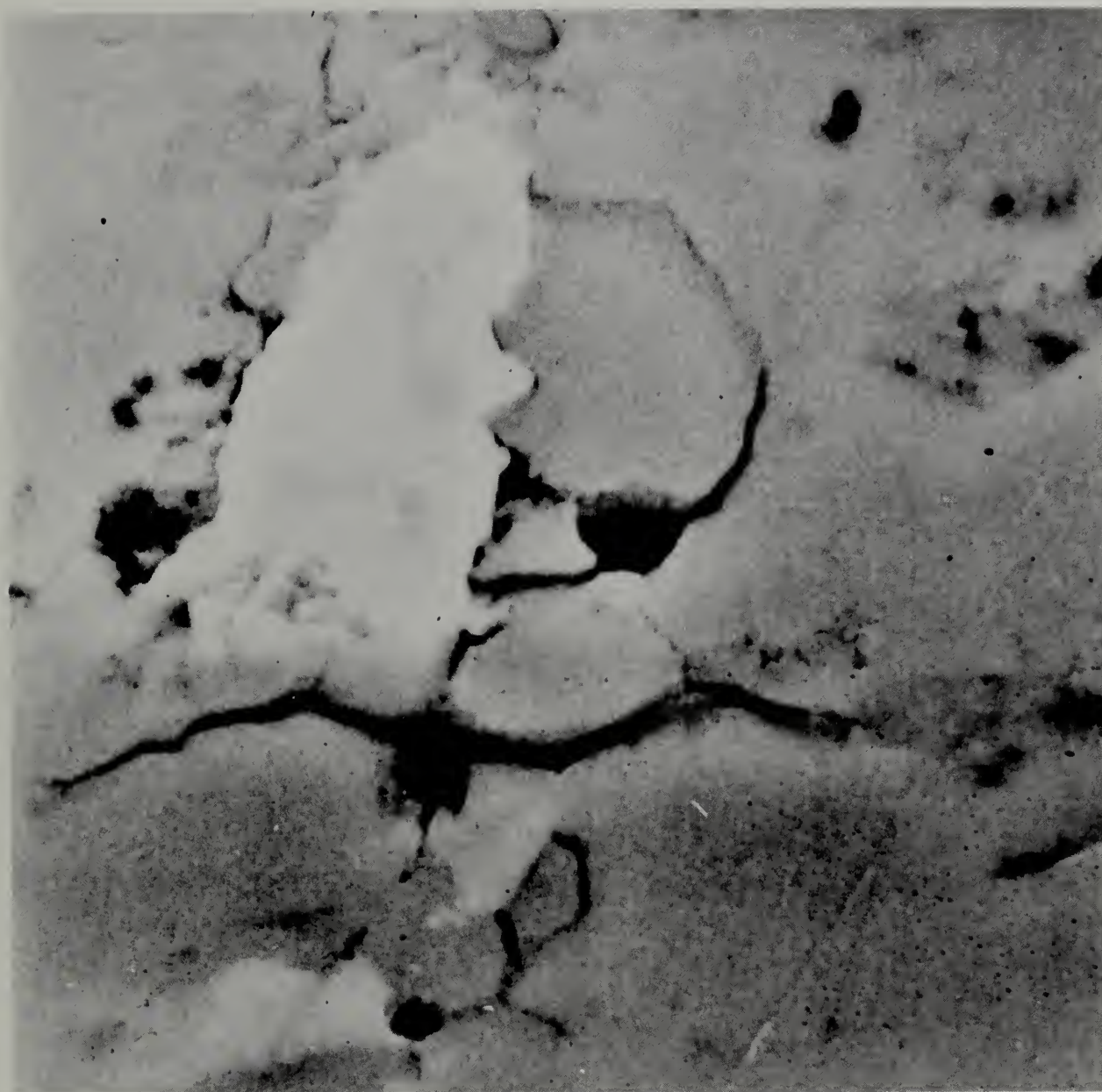
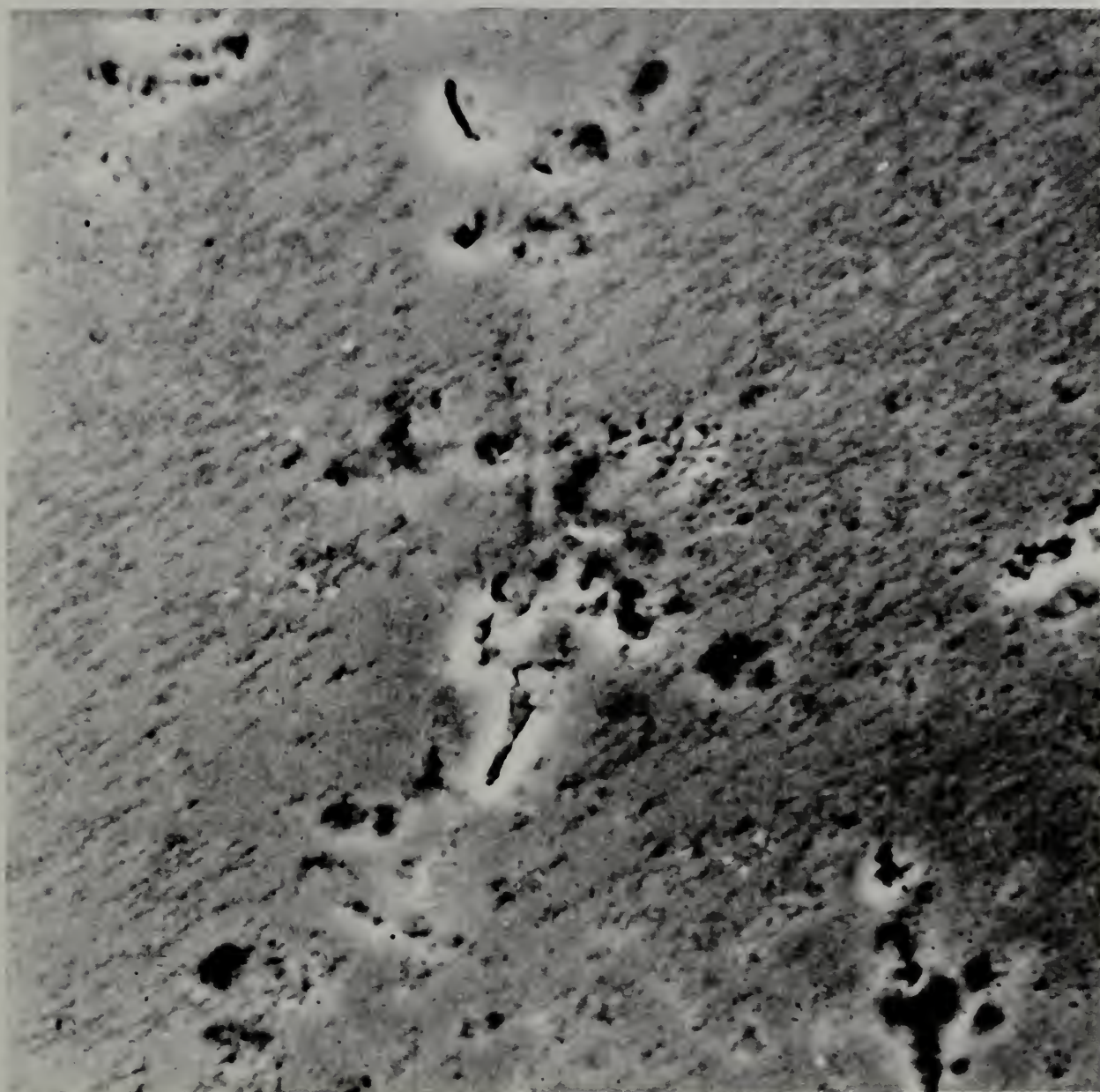


Figure 18. SEM micrograph of longitudinally stressed specimen after 16 hours exposure to chromate solution. The general condition of the specimen was similar to that of a specimen after 7 min. exposure with little additional crack propagation. X4,500



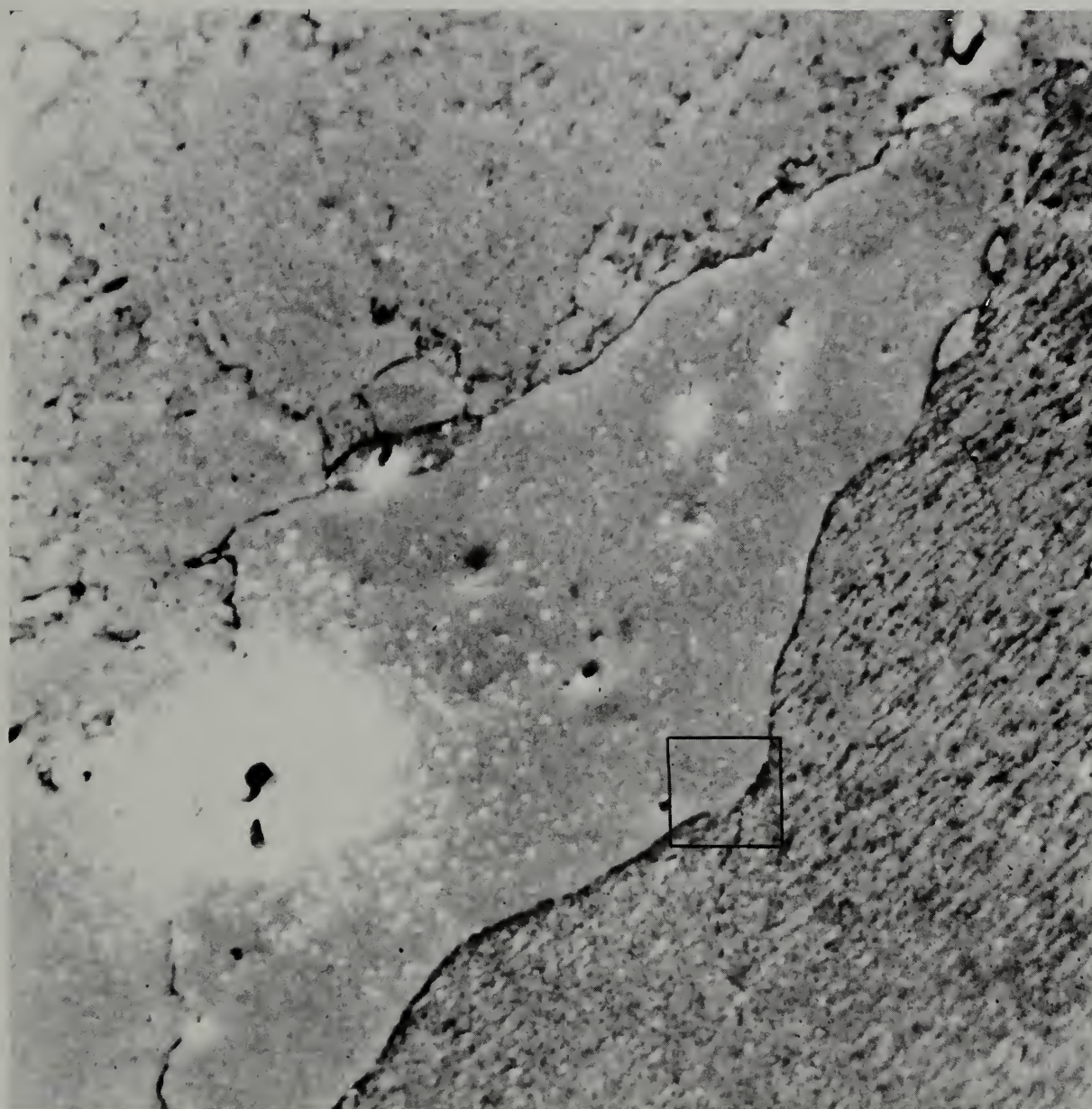


10 $\mu$

Figure 19. SEM micrograph of surface of unstressed specimen exposed to chromate solution for 10 seconds. Note a large amount of general pitting as compared to specimens corroded under stress. X1,500





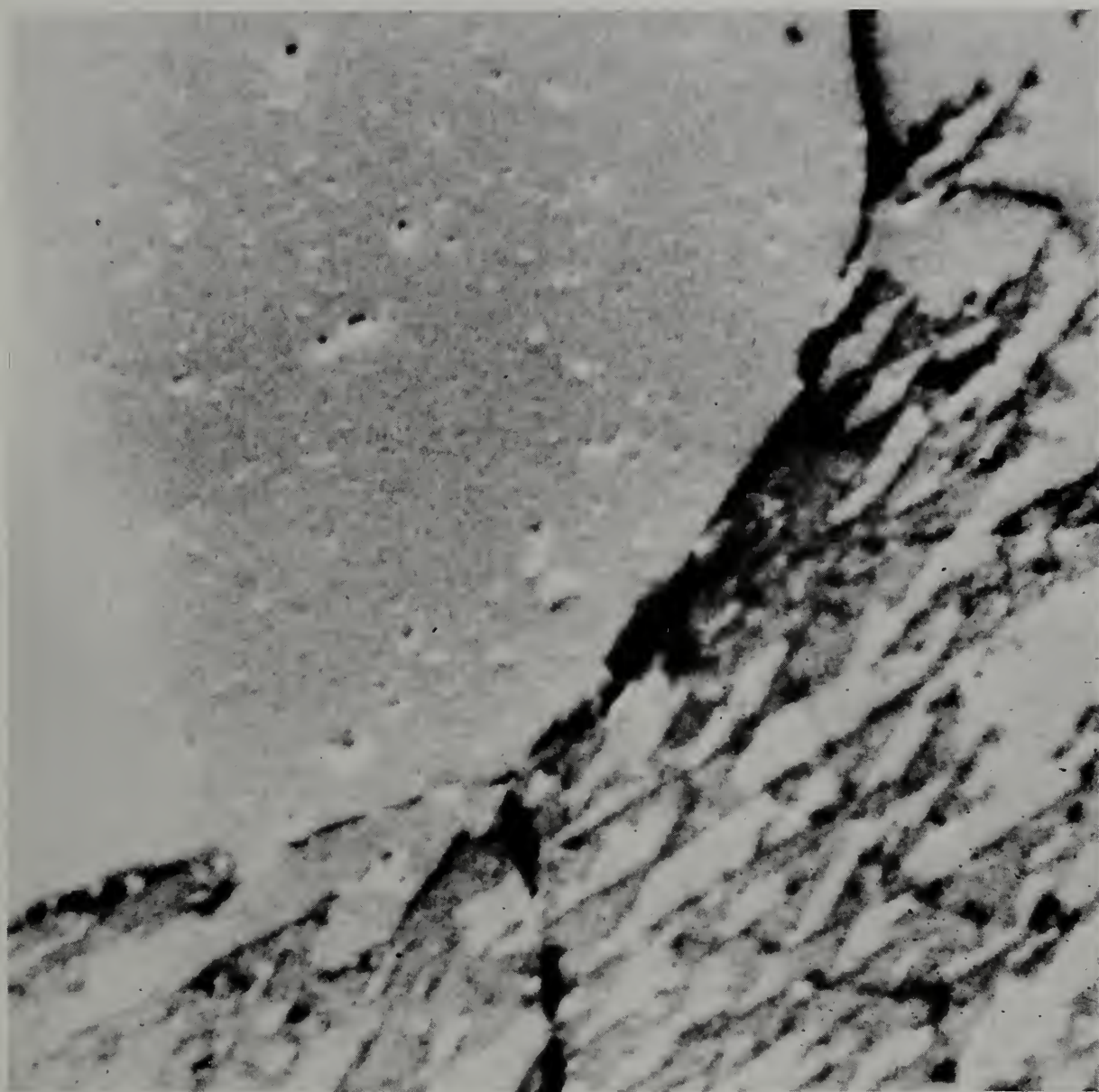


10 $\mu$

Figure 20. SEM micrograph of surface of unstressed specimen exposed to chromate solution for 5 min. Note what appears to be cracks at the grain boundaries at this magnification. See Figure 21 for greater detail along boundaries. X1,500







1μ

Figure 21. SEM micrograph of area marked in Figure 20 after 5 min. exposure to chromate solution shows general grain boundary etching rather than cracking.  
X15,000







